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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF PHOTOVAC MicroFID HANDHELD FLAME IONIZATION DETECTORS AGAINST CHEMICAL WARFARE AGENTS SUMMARY REPORT

Terri L. Longworth
Jacob L. Barnhouse
Kwok Y. Ong
Marcia A. Johnson

ENGINEERING DIRECTORATE

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This report characterizes the chemical warfare (CW) agent detection potential of the commercially available Photovac MicroFID Handheld Flame Ionization Detector. These detectors were tested against HD, GB, and GA vapor at various conditions. This report is intended to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of these detectors.

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PREFACE

The work described herein was authorized under the Expert Assistance (Equipment Test) Program for the U.S. Army Soldier and Biological Chemical Command (SBCCOM) Program Director for Domestic Preparedness. This work was started in December 1998 and completed in January 1999.

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CONTENTS

1. INTRODUCTION 1

2. OBJECTIVE 1

3. SCOPE 1

4. EQUIPMENT AND TEST PROCEDURES 2
4.1 Detector Description 2
4.2 Calibration 3
4.3 Agent Challenge 3
4.4 Agent Vapor Concentration Quantification 4

5. RESULTS AND DISCUSSION 5
5.1 Minimum Detectable Levels 5
5.2 Response Factors 5
5.3 Relative Humidity Effects 6

6. CONCLUSIONS 7

LITERATURE CITED 9

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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF PHOTOVAC MicroFID HANDHELD FLAME IONIZATION DETECTORS AGAINST CHEMICAL WARFARE AGENTS - SUMMARY REPORT

1. INTRODUCTION

In 1996, responding to Public Law 104 - 201, the Department of Defense (DOD) formed the Domestic Preparedness (DP) Program. One of the objectives is to enhance federal, state and local capabilities to respond to Nuclear, Biological and Chemical (NBC) terrorism incidents. Emergency responders who encounter a contaminated or potentially contaminated area must survey the area for the presence of toxic or explosive vapors. Presently, the vapor detectors commonly used are not designed to detect and identify chemical warfare (CW) agents. Little data are available concerning the ability of these commonly used commercially available detection devices to detect CW agents. Under the Domestic Preparedness (DP) Expert Assistance (Test Equipment) Program, the U.S. Army Soldier and Biological Chemical Command (SBCCOM) established a program to address this need. The Design Evaluation Laboratory (DEL) at Aberdeen Proving Ground, Edgewood, Maryland, performed the detector testing.

2. OBJECTIVE

The objective of this test was to assess the capability of the Photovac MicroFID to detect chemical warfare agent vapors. MicroFID is one of several types of chemical detectors selected for evaluation during 1999.

3. SCOPE

The scope of this evaluation was to characterize the CW agent vapor detection capability of the MicroFID detector. The agents used included Tabun (GA), Sarin (GB), and Mustard (HD). These were chosen as representative CW agents because they are believed to be the most likely threats. Test procedures followed those described in the Phase 1 Test Report.[1] The test concept was as follows:

a. For each selected CW agent determine the minimum concentration levels (Minimum Detectable Level, MDL) where repeatable detection readings are achieved. Use Joint Services Operational Requirements (JSOR) as a guide for MDL objectives.

b. Determine the Response Factors (RF) for each CW agent at ambient temperature and low RH.

c. Investigate the effects of humidity and temperature upon the detector response.

d. Observe the effects of potential interference vapors upon detector performance, in the laboratory and in the field.

The test was truncated after the MDL, RF, and humidity effects measurements indicated that the detector responses to CW agents were inaccurate and inconsistent at ambient temperature. Further testing at other temperatures and with interferents was omitted.

4. EQUIPMENT AND TEST PROCEDURES

4.1 Detector Description.

The Photovac MicroFID Handheld Flame Ionization Detector is manufactured by Perkin-Elmer Corporation. This detector was selected for testing based on a Battelle Memorial Institute (BMI) survey[2] that identified the detectors most likely to be used by the local responders in the event of a terrorist incident involving CW agent(s). The devices tested were new and evaluated in the "as received" condition. No attempt was made to optimize their chemical agent detection capability. No pre-test theoretical assessment was made on the detectors except to learn the operating procedures from the manufacturer's User Manual[3]. Conclusions are based solely on the results of CW agent detection during this testing. Aspects of the detectors, other than those described above, were not investigated.

Figure 1 is a digital photograph of a MicroFID. Three were purchased for the evaluation. One unit had an internal electrical malfunction and could not be used in the test. During testing, 110 V AC adapters were used to ensure that detector performance would not be affected by battery condition.

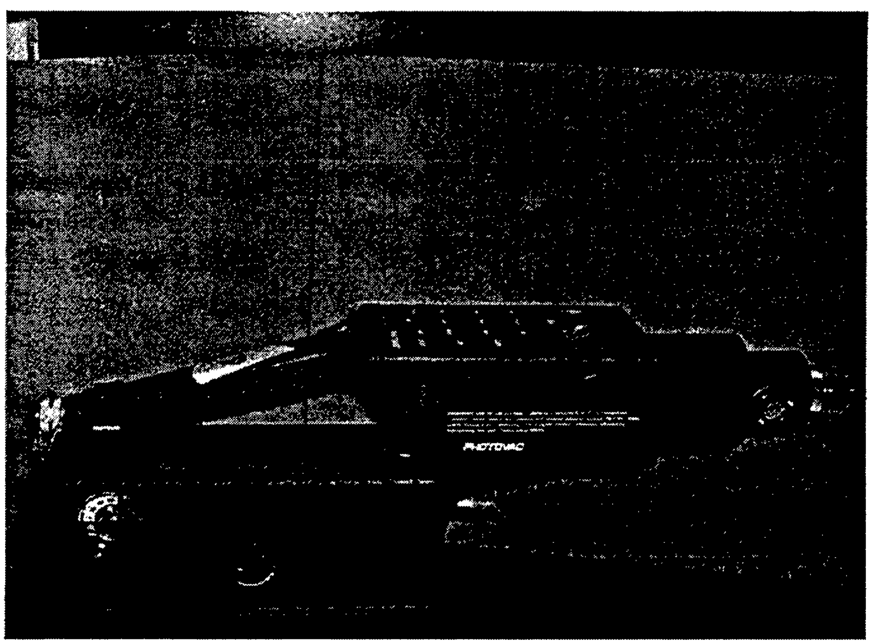

Figure 1. MicroFID

4.2 Calibration.

Each detector was allowed to stabilize before initiating the calibration procedures. Ultra-high purity hydrogen was used to fill the built-in cylinder to provide a consistent flame. Inlet filters were changed according to user manual instructions. Calibrations were performed daily per instructions provided with the detector. Calibration allows the detector to display concentration in parts per million (PPM) units equivalent to the calibration gas. The calibration procedure requires adjusting the detector baseline zero point by challenging with "zero air" ($\leq$ 0.1 PPM total hydrocarbons). Then the detector is challenged with a calibration "span gas" to set the sensitivity. The span gas used was 499.7 PPM of methane in air. Occasionally, 100 PPM of methane in air was used to check the adequacy of the calibration. The detector was re-calibrated if the 100 PPM reading was not within 10% of this expected value. This confirms that the detector was accurately reading methane even when no response readings were seen during a CW agent challenge or when ambient air readings were greater than the CW agent challenge readings.

4.3 Agent Challenge.

The agent challenges were conducted using the Multi-Purpose Chemical Agent Vapor Generation System[4] with zero air and CASARM-grade (high purity) CW agents. The vapor generator permits preconditioning of a detector with humidity-conditioned and temperature-conditioned air prior to challenging it with similarly conditioned air containing the CW agent. Occasionally, the detectors were re-checked with the 100 PPM methane calibration gas after the agent challenges to observe residual effects and/or calibration drift.

Agent testing followed successful detector calibration. First, conditioned air at the desired temperature and humidity from the vapor generator system was sampled by the MicroFID for approximately one minute to establish the stable "background reading" of the detector for the air at each condition. This background reading (baseline) at the testing condition is required to establish the net detector response from the agent challenge reading. The net detector reading is the challenge reading minus the background reading.

Agent challenge begins when the vapor generation system solenoids are energized to switch the air streams from conditioned air only to similarly conditioned air containing the agent. Each detector was tested three times under each condition. The agent challenge time was approximately 5 minutes to allow the maximum detector response. Detector response readings were recorded every minute during the agent challenge. Also, the times for clear down back to the baseline after the agent challenge were noted.

The MicroFID detectors were each tested with the agents GA, GB and HD at different concentration levels at ambient temperature and 0% relative humidity in an attempt to determine the minimum detectable level (MDL) and establish a response curve. The detectors were also tested at other relative humidity conditions (50% and 90%) to observe humidity effects.

The detector response in PPM was observed and recorded. The response readings are relative to the calibration gas. Therefore, the observed detector reading is in PPM methane equivalent units. Response factor (RF) is an indication of the relative sensitivity of a detector to the concentrations of a compound vapor at each condition compared to the calibration gases used. RF is required by MicroFID (Flame Ionization Detector) users to determine the combustible organic compounds in the sample which are ionized by the flame. The displayed reading shows the total concentration of all ionized compounds in the sample. Calculated RFs are commonly entered into a detector's memory to enable the instrument to automatically display the correct concentration readings. The RF is calculated by dividing the actual concentration in the test air (in PPM) by the net detector reading (in PPM).

$$\text{Response Factor} = \frac{(\text{Actual Challenge Concentration})}{(\text{Detector Reading}) - (\text{Background Reading})}$$

Hence, methane will have a response factor of $\frac{499.7 \text{ PPM}}{(499.7 \text{ PPM} - 0 \text{ PPM})} : 1.0.$

Ideally, a compound's RF values should be constant for a given detector model. Higher RF values indicate the detector is less sensitive in detecting that compound vapor.

4.4 Agent Vapor Concentration Quantification.

The generated agent vapor concentrations were analyzed independently and reported in mg/m^3. The measured agent vapor concentration was converted into PPM units for the RF calculations.

The generated agent vapor was quantified by manual sample collection methodology[5] using the Miniature Continuous Air Monitoring System (MINICAMS) manufactured by O. I. Analytical, Inc., Birmingham, Alabama. The MINICAMS is equipped with a flame photometric detector (FPD). This system normally monitors air by collection through sample lines and subsequently adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located after the MINICAMS inlet. Here the concentrated sample is periodically heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification.

For manual sample collection, the PCT was removed from the MINICAMS and connected to a measured suction source to draw the vapor sample from the agent generator. The PCT was then re-inserted into the MINICAMS for analysis. This "manual sample collection" procedure eliminates potential loss of sample through sampling lines and the inlet assembly in order to use the MINICAMS as an analytical instrument. The calibration of the MINICAMS is performed daily using the appropriate standards for the agent of interest.

5. RESULTS AND DISCUSSION

5.1 Minimum Detectable Levels.

The minimum detectable level (MDL) for the MicroFID detectors (A and B) are shown in Table 1 for each agent at ambient temperature and zero percent relative humidity (RH). The 0% RH condition was used to establish the MDL because the detectors were zeroed and calibrated using zero (dry) air.

The MDL concentrations are expressed in mg/m^3 with equivalent parts per million (PPM) values given in parentheses. No meaningful MDL could be established for HD detection. On some occasions the detectors showed readings of approximately 0.5 PPM. Although that might be construed as a "detection," later readings remained below 0.5 even at the highest agent concentrations tested. Those results are identified in the table with the asterisk (*) to indicate that at these concentration levels minimal or no response was observed.

The Joint Service Operational Requirements (JSOR) for CW agent sensitivity for point detectors are also listed to show the relatively low detection sensitivity of the MicroFID detectors compared to the JSOR requirements. All MDLs were considerably higher than the JSOR values.

Table 1. MDL at Ambient Temperature and 0% Relative Humidity

Minimum Detectable Level, mg/m^3 (PPM)			
AGENT	Detector A	Detector B	JSOR
HD	>46 (7.03)*	>23.5 (3.59)*	2 (0.3)
GA	13.9 (1.30)	13.9 (1.30)	0.1 (0.017)
GB	27.8 (4.84)	27.8 (4.84)	0.1 (0.017)

*Not measurable or minimal detector response observed up to this concentration level

5.2 Response Factors.

Response factors (RF) for the CW agents tested were calculated at ambient temperatures (19-25°C) and the relative humidity conditions of 0, 50 and 90%. The ranges of calculated RF values at the average temperature and humidity conditions are summarized in Table 2. The RF values listed represent the results of multiple challenges at agent concentrations between 3 and 60 mg/m^3. No direct relationship between RF and agent concentration was observed.

Ideally, the RF values for both detectors should be very similar. However, these detectors were found to give varied and inconsistent values when exposed to similar concentrations of CW agent vapors. For example, one detector would be more sensitive than the other to a particular test condition on one day, then the reverse would be observed on another day, even when both detectors were repeatedly responding similarly and correctly to the calibration gas challenges.

Results consolidated in Table 2 reflect the wide ranges of RFs observed at different concentrations and conditions for the detectors tested. It presents the highest and the lowest RF calculated from results of each tested condition. The highest RFs reported as NR (No Response) means the detector failed to respond even at concentrations much higher than the JSOR requirements. Essentially, this gives a value of zero for the denominator of the RF equation causing the RF to go to infinity. No consistent response factor for any detector or condition could be determined.

The detectors were frequently rechecked for their performance with the methane calibration gas to observe residual effects and/or calibration drift and to assure validity of the inconsistent behaviors. The detector responses to these challenges suggest that the detector sensitivity was not degraded by exposure to CW agent vapor. Calculating the response factors on the 100 PPM methane gas challenges recorded during all testing for both detectors shows a RF range of 1.04 to 1.13. In contrast, the RFs recorded during the CW agent tests shown in Table 2 show no consistency between detectors A and B, and an unacceptable range between low and high values for all agents tested. Observations suggest that these detectors cannot be relied upon for CW agent vapor detection and warning.

Table 2. Range of Response Factors at 20°C and Various RH Conditions

CW Agent	Average Temperature °C	Relative Humidity %	Detector A		Detector B	
			Lowest RF	Highest RF*	Lowest RF	Highest RF*
HD	20	0	6.41	NR	5.98	NR
HD	20	50	23.29	93.19	9.32	18.63
HD	20	90	90.1	NR	11.26	15.02
GA	20	0	0.7	NR	1.89	NR
GA	20	50	1.33	1.49	1.95	2.82
GA	20	90	1.0	1.14	0.8	1.33
GB	20	0	4.4	NR	4.03	NR
GB	20	50	2.52	19.08	2.69	8.18
GB	20	90	4.46	NR	4.78	NR

* NR indicates no meaningful detector response observed

5.3 Relative Humidity Effects.

The MicroFID baseline response did not appear to be affected by relative humidity changes. All baseline readings were zero at the different humidity conditions tested even though the detectors were calibrated with zero (dry) air only. No conclusive RH effects could be determined due to the large ranges and inconsistencies of the detector readings.

6. CONCLUSIONS

The current JSOR is 0.1 mg/m^3 for the G (nerve) agents and 2 mg/m^3 for the H (blister) agents. CW agent challenges to the Photovac MicroFID Handheld Flame Ionization Detector showed that they were not sensitive enough to detect CW agents at concentrations within an order of magnitude of the JSOR levels for any of the conditions tested. The MicroFID responded only to very high concentrations of CW agents.

The unpredictable CW agent detection performance prevented the establishment of a reliable response curve. Test results suggest that the MicroFID in its current configuration cannot be used effectively for CW agent detection.

Recalibration checks clearly suggest that the detector sensitivity is not degraded by exposure to CW agent vapor as methane detection capability was nearly constant. Methane detection responses also did not appear to be affected by relative humidity changes. However, CW agent detection at varied humidity conditions showed gross variance.

Testing was discontinued after the ambient temperature agent sensitivity tests based on the test results that indicated poor performance toward CW agent detection. Further testing of the detectors at other planned conditions was considered to be of no value. It can be concluded that the MicroFID detectors will not provide a dependable means of detecting the presence of CW agent vapors.

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